

CELL ANALYSIS, MODELING, AND PROTOTYPING (CAMP) FACILITY RESEARCH ACTIVITIES

Project ID: ES030

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2017 U.S. DOE HYDROGEN and FUEL
CELLS PROGRAM and VEHICLE
TECHNOLOGIES OFFICE ANNUAL MERIT
REVIEW AND PEER EVALUATION MEETING

Washington, D.C.

June 5-9, 2017

OVERVIEW

Timeline

- Start: October 1, 2014
- Finish: September 30, 2018

Budget

- \$1,900 K for FY17
 - 100% DOE-ABR

Barriers

- Need a high energy density battery for Electric Vehicle (EV) use that is safe, cost-effective, has long cycle life, and meets or exceeds DOE/USABC goals.
 - Independent validation analysis of newly developed battery materials are needed in cell formats with at least 0.2 Ah before larger scale industrial commitment

Partners

- SNL
- NREL
- Coordinated effort with DOE-EERE-VTO Next Generation Anodes and HE-HV Projects (ANL, LBNL, SNL, ORNL, NREL)
- Argonne Facilities: MERF, EADL, CNM & PTF
- See Collaboration list at end

RELEVANCE/OBJECTIVES

- Transition new high energy battery chemistries invented in research laboratories to industrial production through independent validation and analysis in prototype cell formats (xx3450 & xx6395 pouch cells & 18650 cells) with 20 to 3,000 mAh capacity.



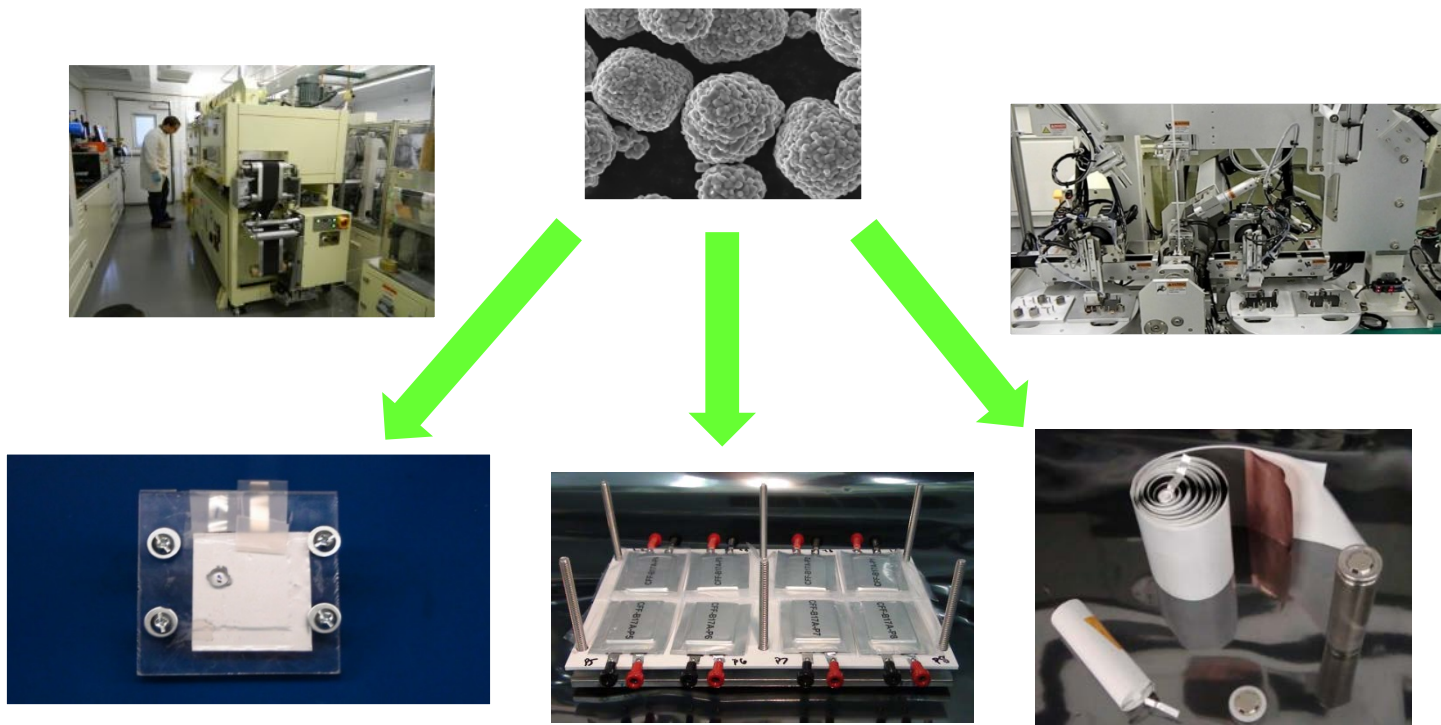
- Researchers are often not able to provide the quantities of novel materials needed to make a full size EV cell to demonstrate the merits of their discoveries. The CAMP Facility is ideally sized to explore new materials with quantities as small as 50 grams for active materials, and even less for electrode/electrolyte additives.

FY17 PROGRESS MEASURES & MILESTONES

Milestone	Planned End Date	Type	Status
Assess performance of full cells with LiFSI and LiTDI-based electrolytes using multiple electrochemical and physicochemical characterization techniques (FY16)	9/30/2016	Milestone	Completed
Fabricate pouch cells with at least 400 mAh based on latest improvements to silicon-composite negative electrode developed in CAMP and NMC positive electrode (FY16)	9/30/2016	Milestone	Completed
Produce 20 meters of advanced double-sided anode and cathode matching electrodes for SiLion SBIR Phase II project	11/11/2016	Quarterly Progress	Completed
Deliver at least 10 meters of alumina-coated NMC532 single-sided electrodes to "High Energy/High Voltage" project	12/22/2016	Quarterly Progress	Completed
Deliver to "Next Generation Anodes" project at least 10 meters of single-sided negative electrode based on 30% silicon-graphite with ~ 2 mAh/cm ² loading for FY17 baseline	3/31/2017	Quarterly Progress	Delayed 3 weeks
Develop techniques for in-operando detection of Li-plating during fast charging	5/26/2017	Quarterly Progress	On-going
Determine effect of cycle life/calendar life/cell pressure on silicon-graphite vs. NMC532 pouch cells	6/30/2017	Quarterly Progress	On-going
Fabricate 30 pouch cells with >400 mAh capacity using Si-Graphite anode and NMC cathode for NREL	6/30/2017	Milestone	On-going
Submit final report on advanced battery materials validated in FY17 and summary of Electrode Library activity	9/29/2017	Quarterly Progress	On-going
Fabricate pouch cells with at least 1 Ah capacity based on latest improvements to composite negative electrode developed in CAMP and high nickel NMC positive electrode	9/29/2017	Milestone	On-going

APPROACH/RESOURCES

- Researchers submit materials with promising energy density
 - Small hand-coated electrodes are made
 - Coin cells are made and tested
- } Glove box
Benchtop
- Larger material samples are obtained (MERF, partnerships, etc.)
 - Longer lengths of electrode are made from scaled materials
 - Pouch cell or 18650s are made and tested
- } Dry Room
Pilot scale
- Extensive diagnostics & electrochemical modeling on promising technologies

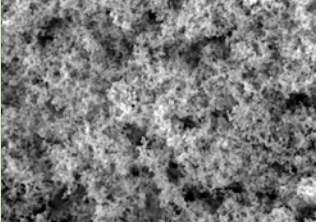
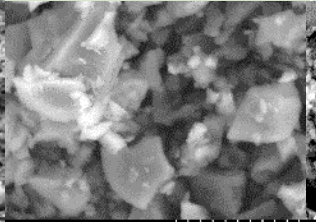
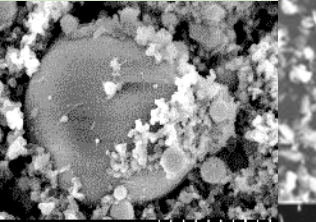
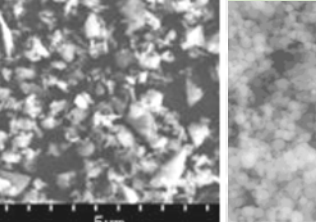
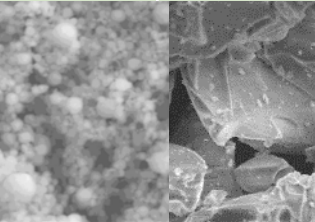
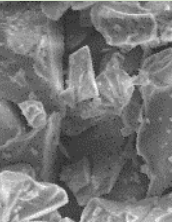


TECHNICAL ACCOMPLISHMENTS

- Continued development of silicon-graphite cells with new silicon sources
- Initiated study on effects of calendar vs. cycle life on silicon-graphite//NMC pouch cells
- Supplied numerous baseline electrodes, prototype electrodes & cells for High-Energy High-Voltage and Next Generation Anodes Projects ([see ES252, ES253, ES254, ES261, ES262](#))
- Coated and tested various Al_2O_3 coatings on NMC532 cathode particles ([see ES254](#))
- Supplied data, electrodes and pouch cells for NREL's CAEBAT projects ([see ES298, ES299](#))
- Examined performance of lithium-ion cells with LiFSI and LiTDI electrolytes
- Fabricated electrodes in support of SiLLion's DOE SBIR Phase II project
- Studied phase separation and polymer aggregation of FEC-LiPF₆ electrolytes
- Supported conductive binder development with Argonne's MERF and LBNL ([see ES168](#))
- Fabricated calendered and uncalendered electrodes for NREL's ALD coating scale up
- Fabricated and tested multi-layer pouch cells for continued study of the DOE-EERE Award-Zhang (high voltage electrolyte)
- Collaborated with various separator manufactures by supplying electrodes and evaluating separators for High-Voltage battery applications
- Collaborated with various silicon manufactures by supplying electrodes and evaluating materials for Next Generation Anodes Project
- Supported numerous DOE projects with prototype electrodes via Electrode Library
- Expanded electrochemical model for interfacial impedance and bulk transport

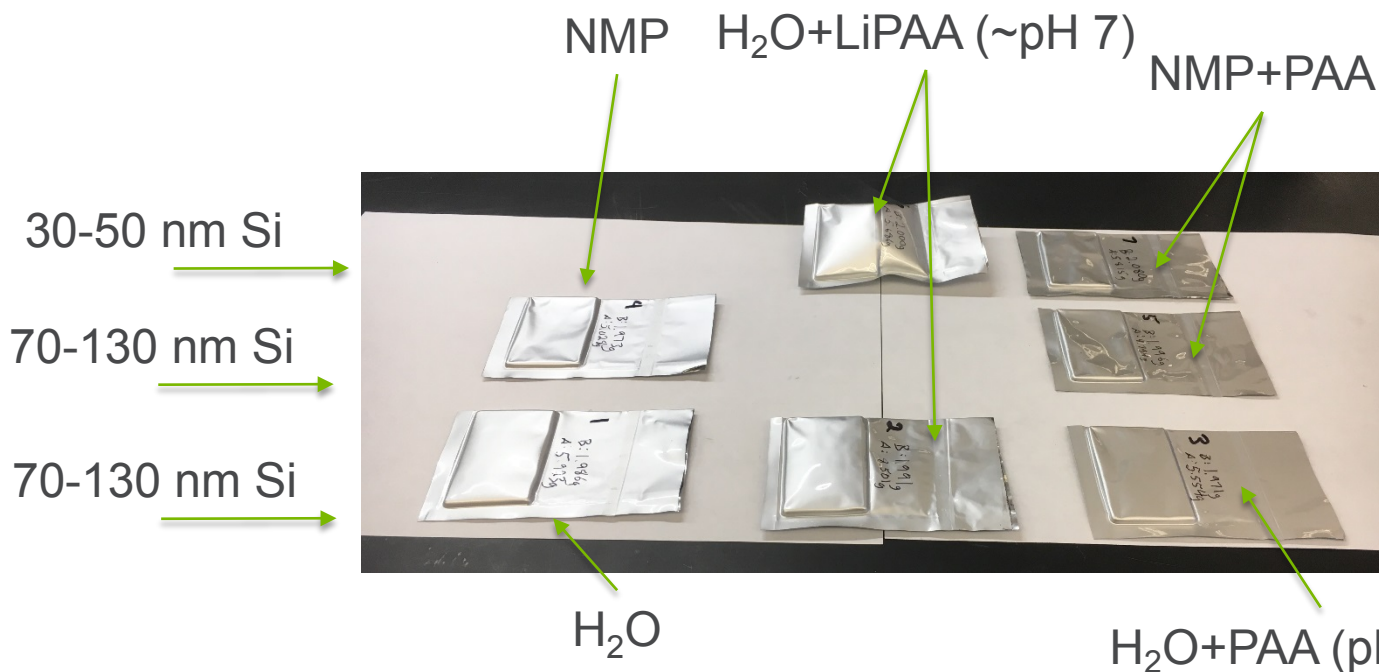
SWITCH TO NEW SILICON POWDER BASELINE

- 50-70 nm silicon from NanoAmor (baseline) discontinued in late 2015
- 30-50 nm silicon sample from NanoAmor coated well on pilot scale coater using formula very similar to 50-70 nm Si. However, two key limitations observed:
 - smaller particle size resulted in excessive SEI and SiO₂ volume
 - ARC tests at SNL indicated poor thermal abuse response for smaller Si
- Decided to move to larger particle size (>70 nm)
- Selected NanoAmor 70-130 nm Si, but after numerous variations, performance at higher loadings (~3.5 mAh/cm²) were not as good as with 50-70 nm Si.
- Still looking for better Si-based materials – now testing Si from Paraclete Energy

NanoAmor	NanoAmor	American Elements	American Elements	Hydro-Quebec	Aldrich
30-50 nm Si	70-130 nm Si	150 nm Si	500 nm Si	80 nm Si	40 μm SiO
Grown	Milled	Grown	Milled	Grown	Grown/milled
					

SILICON POWDERS GENERATE GAS DURING AQUEOUS SLURRY ELECTRODE PROCESSING

- Sealed pouch material containing various slurry components show the most significant swelling for higher surface area silicon with water and LiPAA.
- Note: these are not actual cells (no graphite or carbon; Si/binder slurry only)



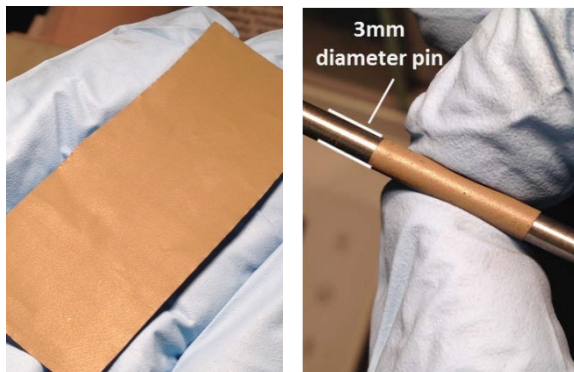
All pouches started with roughly the same shape – pictures show the gassing that occurred after sitting on a counter for 72 hours

NEW 70-130 nm SILICON ELECTRODES SUFFER FROM RESIDUAL BINDER STRESSES

- Replicate baseline electrode processing
 - Using NanoAmor Si 70-130 nm and Hitachi MagE3

Original Baseline Electrodes
A-A006 & A-A006A

NanoAmor Silicon
50 to 70nm, MagE



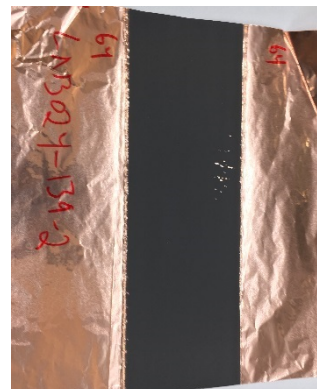
NanoAmor Silicon
30 to 50nm, MagE



Front

Back

NanoAmor Silicon
70 to 130nm, MagE3



Front



Back



NanoAmor Silicon 70 to
130nm,
Prod#: 0143KE,
Lot#: 0143-042916

Items	Unit	MAGE3
Lot Number	-	#160523
Quantity	kg	80
Average Particle Size	μm	22.4
S.S.A.	$\times 10^3 \text{ m}^2/\text{kg}$	3.9
Tap Density	$\times 10^3 \text{ kg/m}^3$	0.90

Hitachi, MagE3 graphite,
Lot#: 160523

Changing composition and MW of binder showed marginal improvements – but all had persistent issues with coating quality at higher loadings ($> 3 \text{ mAh/cm}^2$)

ELECTRODE FABRICATION – TRIAL RUNS WITH 70-130 nm SILICON

Idea	Electrode ID	NanoAmor Silicon 70 to 130nm, %	Hitachi, MagE3 graphite, %	Timcal C45, %	Binder, %	Result
Replicate baseline electrode processing	LN3024-139-2	15	73	2	10 (450k LiPAA)	<u>High stresses observed after drying</u>
Premix of LN3024-139	LN3024-140-2	55.6	0	7.4	37.0 (450k LiPAA)	Thin OK
Graphite only w/ PVDF	LN3024-141-2	0	91.83	2	6 (PVDF) + 0.17 oxalic acid	Good
Increase composition surface area	LN3024-142-2	30	58	2	10 (450k LiPAA)	Minimal improvement
Decrease binder	LN3024-143-2	15	75	2	8 (450k LiPAA)	High stresses
Try 250k LiPAA	LN3024-147	15	75	2	10 (250k LiPAA)	Less stresses but still present
250k LiPAA w/ higher Si and less binder	LN3024-149	30	62	2	6 (250k LiPAA)	Less stresses but still present
250k LiPAA w/ less binder	LN3024-150	15	77	2	6 (250k LiPAA)	Less stresses but still present
250k LiPAA w/ even less binder	LN3024-151	15	79	2	4 (250k LiPAA)	Noticeable decrease in adhesion properties

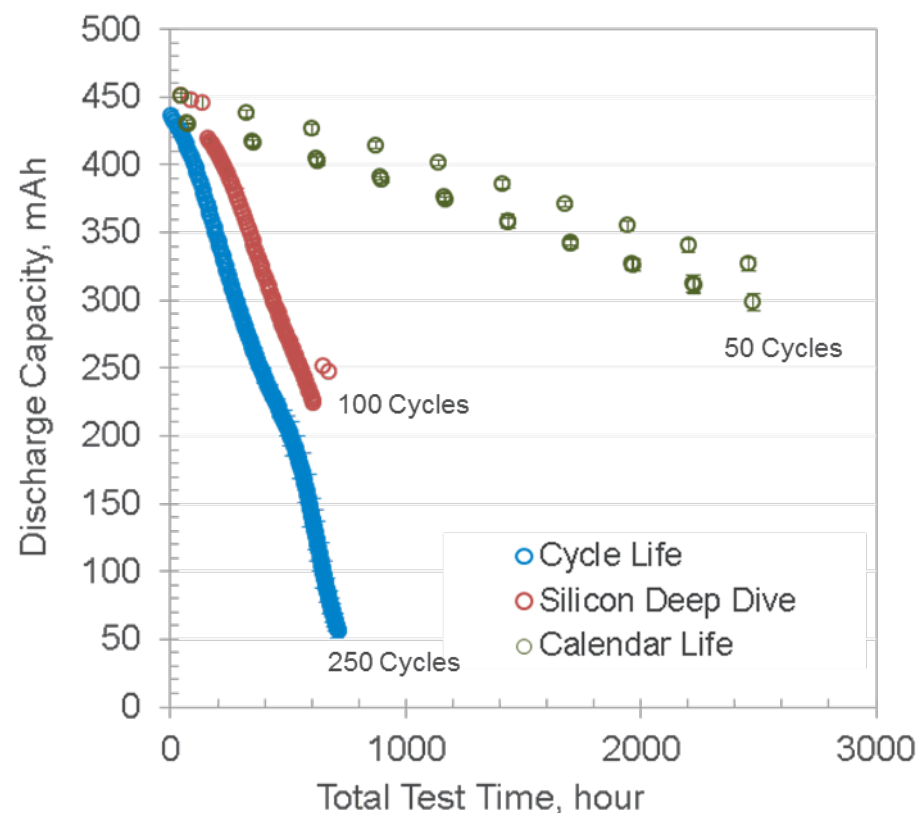
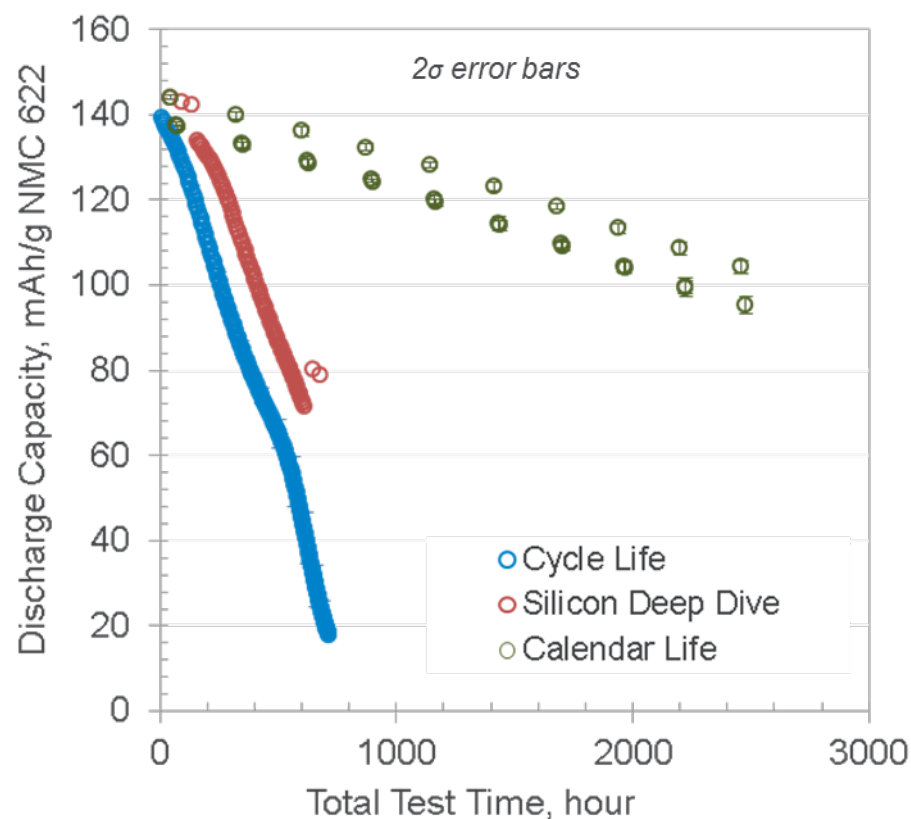
TEST EFFECTS OF CALENDAR VS. CYCLE LIFE IN 450 mAh POUCH CELLS

Evaluate Cycle Life vs Silicon Deep Dive vs Calendar Life Testing

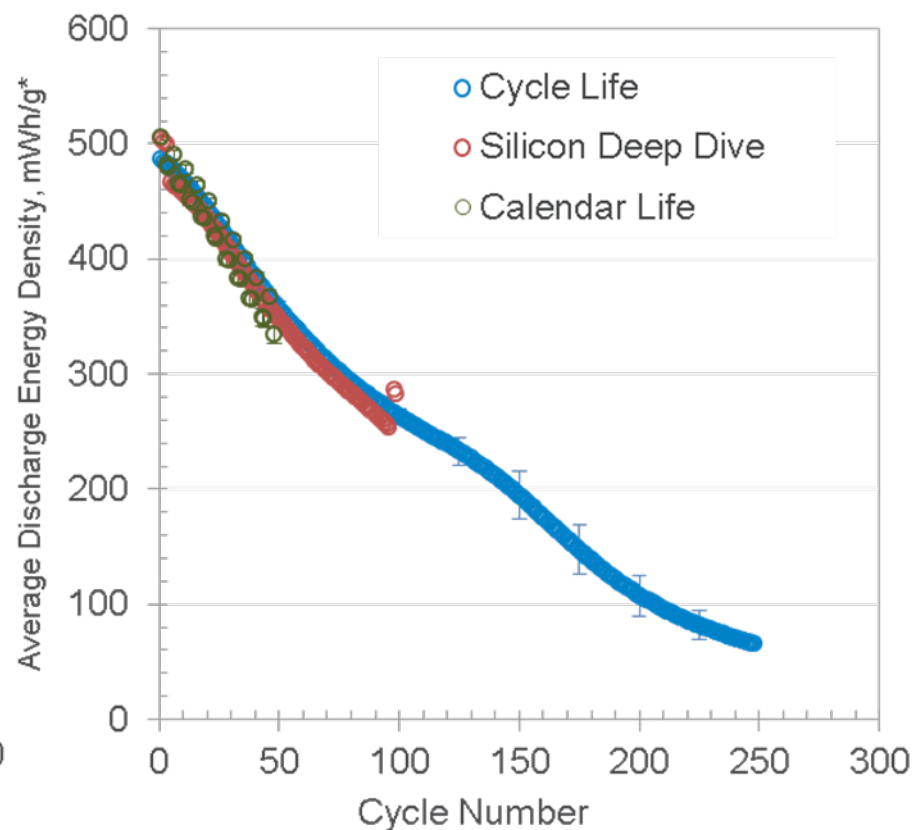
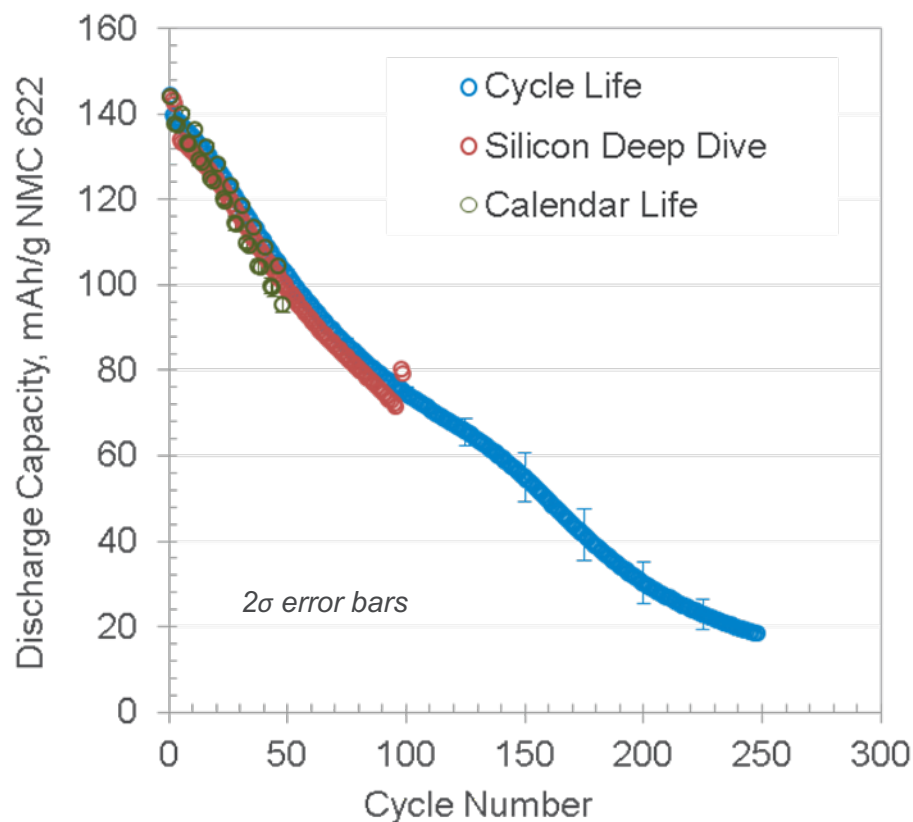
Silicon-graphite (15% Si (50-70nm)) vs. NMC622 using 90wt.%Gen2+10wt.%FEC

- **Formation (same test performed for each set) – all 12 cells**
 - Tap Charge to 1.5V for 15 min., rest 24 hr., 2 cycles @ C/20, 5 cycles @ C/10 discharge and C/10 charge (trickle to C/16), rest 48 hr., **3.0 to 4.1V**
- **Cycle Life – 4 cells**
 - 250 cycle @ C/2 discharge, C/3 charge (trickle to C/20), 3.0 to 4.1V
- **Silicon Deep Dive Protocol (for comparison) – 4 cells**
 - C/20 (no trickle) 3X, HPPC (3C dischrg. and 2.25C chrg.), C/3 discharge and C/3 charge (no trickle) 92X, HPPC, C/20 (no trickle) 3X, 3.0 to 4.1V
- **Calendar Life – 4 cells**
 - C/20 (no trickle) 1.5X, HPPC (3C dischrg. and 2.25C chrg.), then 2 cycles C/3 charge (trickle to C/20) and C/2 discharge, C/20 charge to 3.9V, hold at 3.9V for 3 hours, rest 168 hr (or 7 days), repeat 5 times then hold at 4.0V for 3 hours, rest 168 hr (or 7 days), repeat 5 times, 3.0 to 4.1V

CALENDAR TESTING HAS LESS CAPACITY FADE THAN CYCLE LIFE TESTING WHEN LOOKING AT TIME ONLY



NO DIFFERENCE BETWEEN CALENDAR TESTING AND CYCLE LIFE TESTING WHEN LOOKING AT CYCLES ONLY



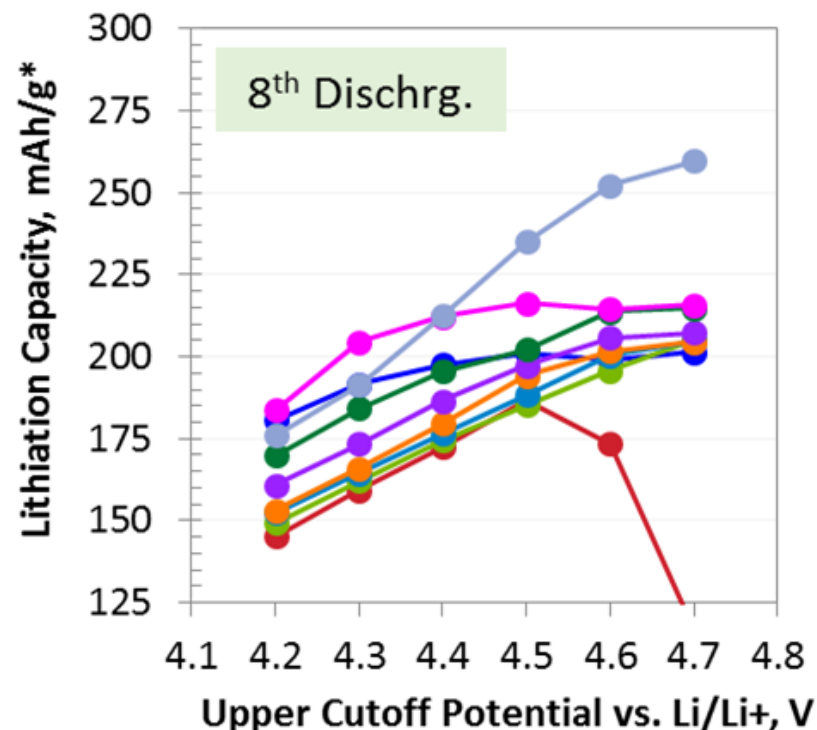
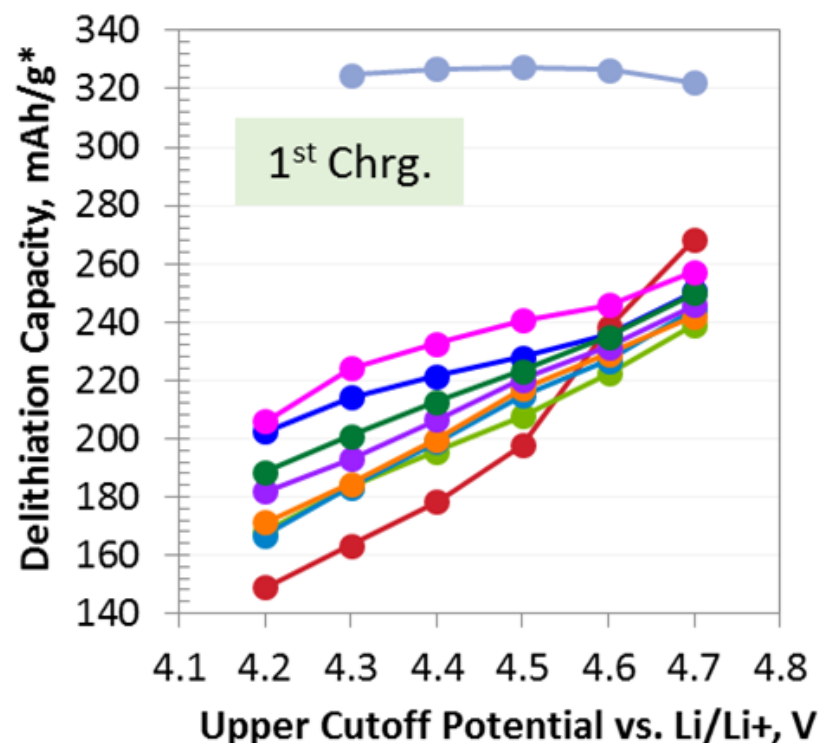
VOLTAGE WINDOW STUDY OF CATHODES TO SUPPORT TECHNO-ECONOMIC MODELING IN BATPAC

- Testing at Upper Cutoff Voltages of: 4.2, 4.3, 4.4, 4.5, 4.6, & 4.7 V vs. Li/Li⁺
 - Cathode: variable
 - Anode: Lithium metal chip (MTI)
 - Electrolyte: 1.2 M LiPF₆ in EC:EMC (3:7wt.%) Tomiyama [Gen2]
 - Half-cell, coin cell, 4 cells for each voltage and for each material at 30°C
 - Total of 24 coin cells tested for each material

(HE5050 is an exception, cells formed from 2.5 to 4.7V, and rate study used 2.5V lower cut off voltage)

Test	Cycles	Cycle # used for data analysis	Description
Formation	3	1st	no tap charge, C/10 rate using a C/24 trickle charge, 2 minute rest between Ch/Disch
Rate Study	2	5th	Charge C/24 (w/o trickle), Discharge C/24 2 minute rest between Ch/Disch
	3	8th	Charge C/10 (w/ C/24 trickle), Discharge C/10 2 minute rest between Ch/Disch
	3		Charge C/5 (w/ C/24 trickle), Discharge C/5 2 minute rest between Ch/Disch
	3		Charge C/5 (w/ C/24 trickle), Discharge C/2 2 minute rest between Ch/Disch
	3		Charge C/5 (w/ C/24 trickle), Discharge 1C 2 minute rest between Ch/Disch
	3		Charge C/5 (w/ C/24 trickle), Discharge 2C 2 minute rest between Ch/Disch
	20 cycles total		

VOLTAGE WINDOW STUDY: CAPACITY, mAh/g

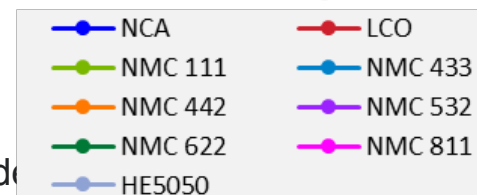
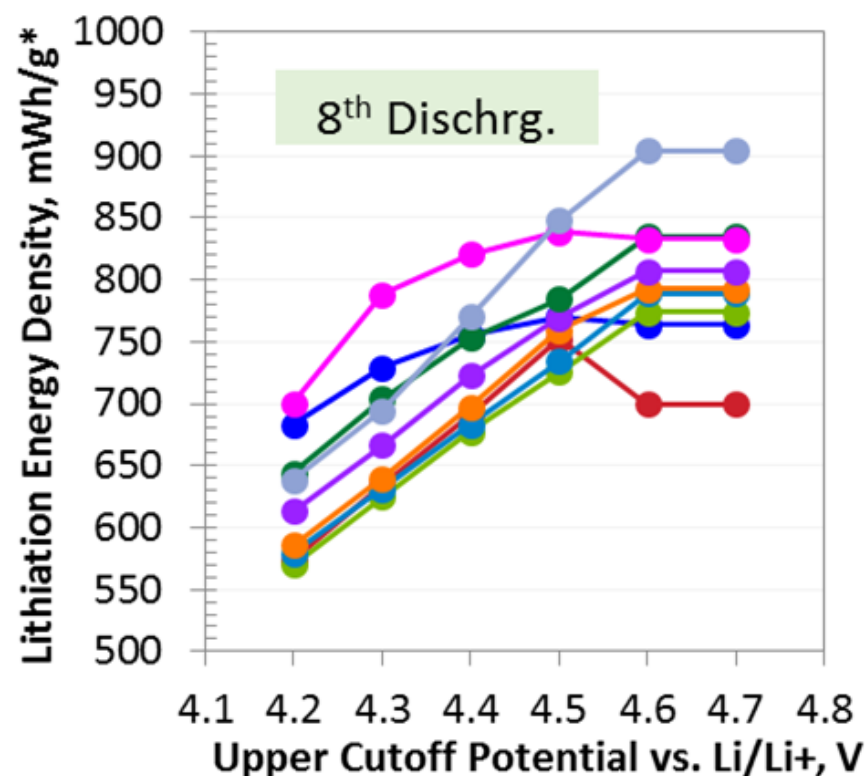
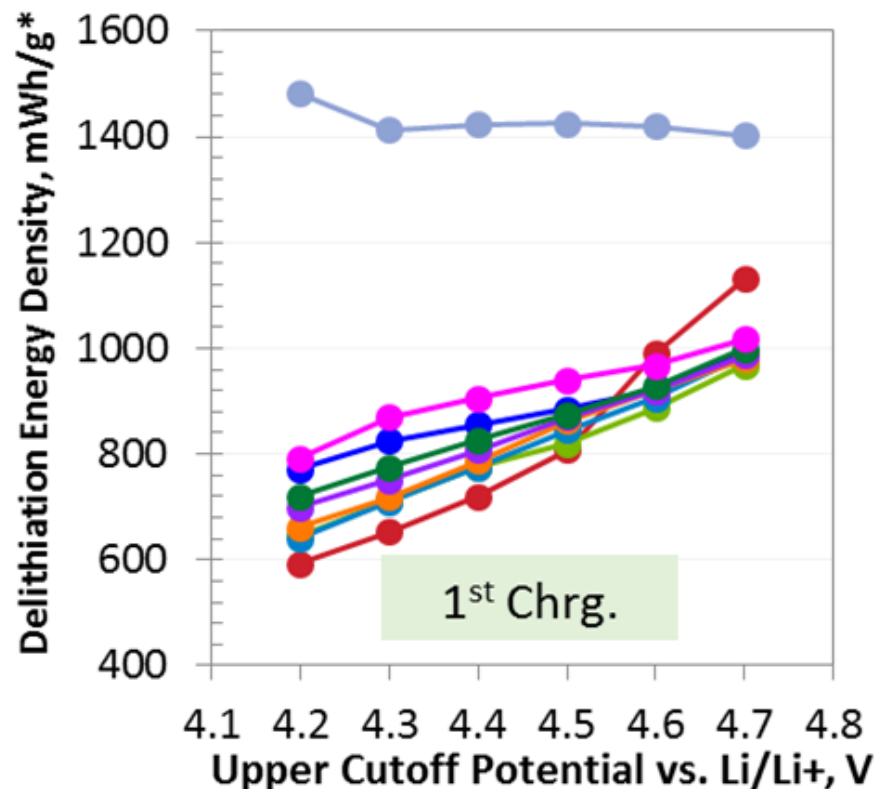


- These values are used to design matching negative-positive electrode pairs
- ≤4.4 V vs. Li/Li⁺, capacity data suggests NCM811 as the preferred cathode
- >4.5 V vs. Li/Li⁺, capacity data suggests NCM622 or NCM811 as the preferred cathode
- While LCO shows a significant increase of capacity when >4.4 V vs. Li/Li⁺ for the 1st delithiation (LEFT), there are known issues of “lattice defects, oxygen loss, transition metal dissolution, and/or structural degradation” at higher voltages[#] -> the degradation of performance at higher voltages is observed during the 8th discharge (RIGHT).

*per gram of oxide

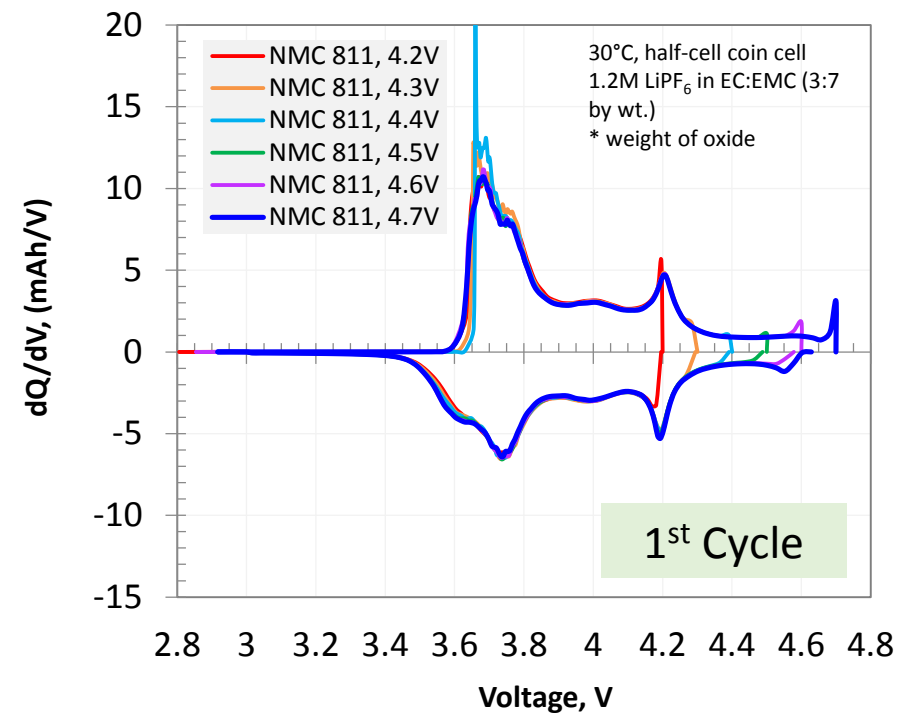
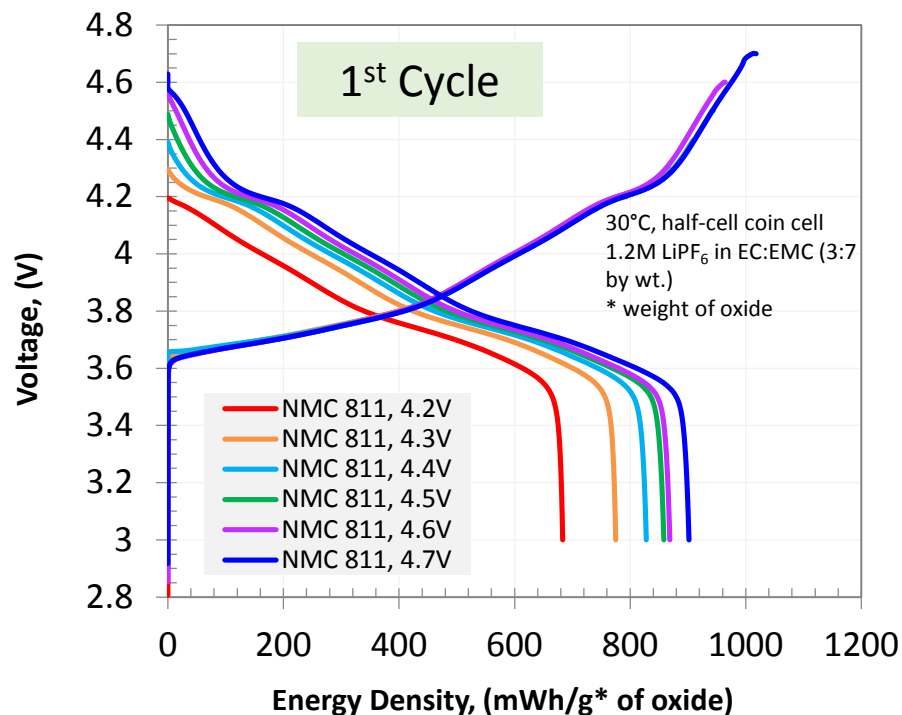
[#]Extending the High-Voltage Capacity of LiCoO₂ Cathode by Direct Coating of the Composite Electrode with Li₂CO₃ via Magnetron Sputtering, Xinyi Dai and et. al. The Journal of Physical Chemistry, 2016, DOI: 10.1021/acs.jpcc.5b10677

VOLTAGE WINDOW STUDY: ENERGY, mWh/g



- ≤ 4.5 V vs. Li/Li⁺, energy density data suggests NCM811 as the preferred cathode
- > 4.6 V vs. Li/Li⁺, energy density data suggests NCM622 or NCM811 as the preferred cathode
- Other properties such as thermal abuse response, cycle life, and cost must be considered.

NEW CATHODE ADDED TO ELECTRODE LIBRARY: NMC811



- These plots show the trends of representative performance for the 1st cycle (~C/10) of NMC811 vs. Li metal as the upper cut off voltage was increased from 4.2V to 4.7V
- Additional electrodes were added to the Electrode Library as materials that are relevant to the DOE-EERE-VTO Energy Storage programs became available

ELECTRODES PRODUCED FOR HE-HV, SILICON DEEP DIVE, AND OTHER DOE FUNDED PROJECTS

From May 2016- to Current:

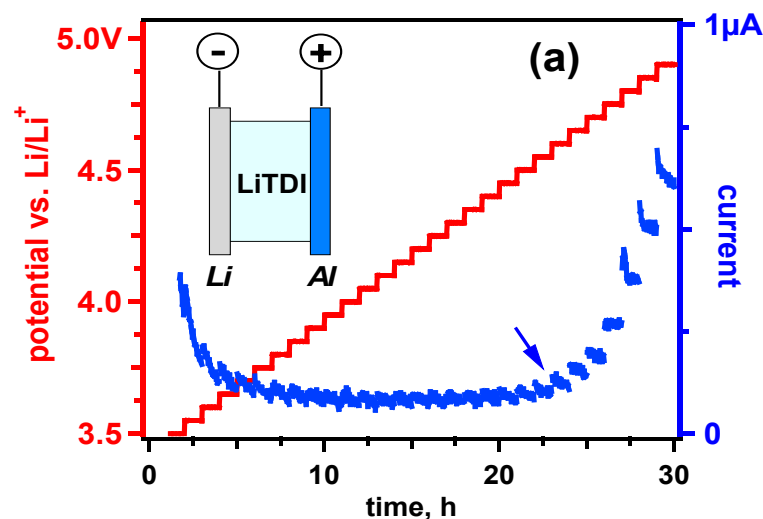
- HE-HV Project
 - 203 Sheets = 4.91 sq. meters
 - Added: NMC811(HV&LV), MCMB
 - Replenished: A12, NMC532(HV)
- Silicon Deep Dive Project
 - 535 Sheets = 12.95 sq. meters
 - Added: New Baseline (In Progress)
 - Replenished: NMC532(LV)
- SBIR/STTR/SBV Projects
 - 511 Sheets = 12.37 sq. meters

(see ES252, ES253, ES254, ES261, ES262)



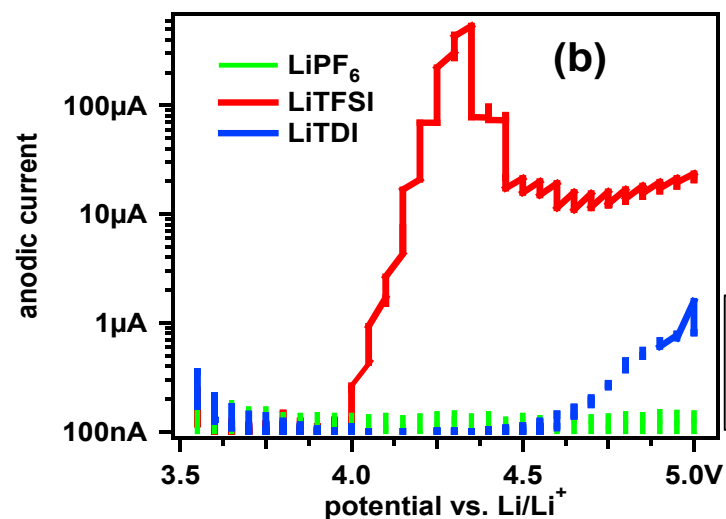
LiTDI CONTAINING ELECTROLYTES DO NOT CORRODE THE AL CURRENT COLLECTOR

Al/Li cells containing 0.5 M LiTDI in EC:EMC (3:7, w/w) electrolyte



Potential and current as a function of time

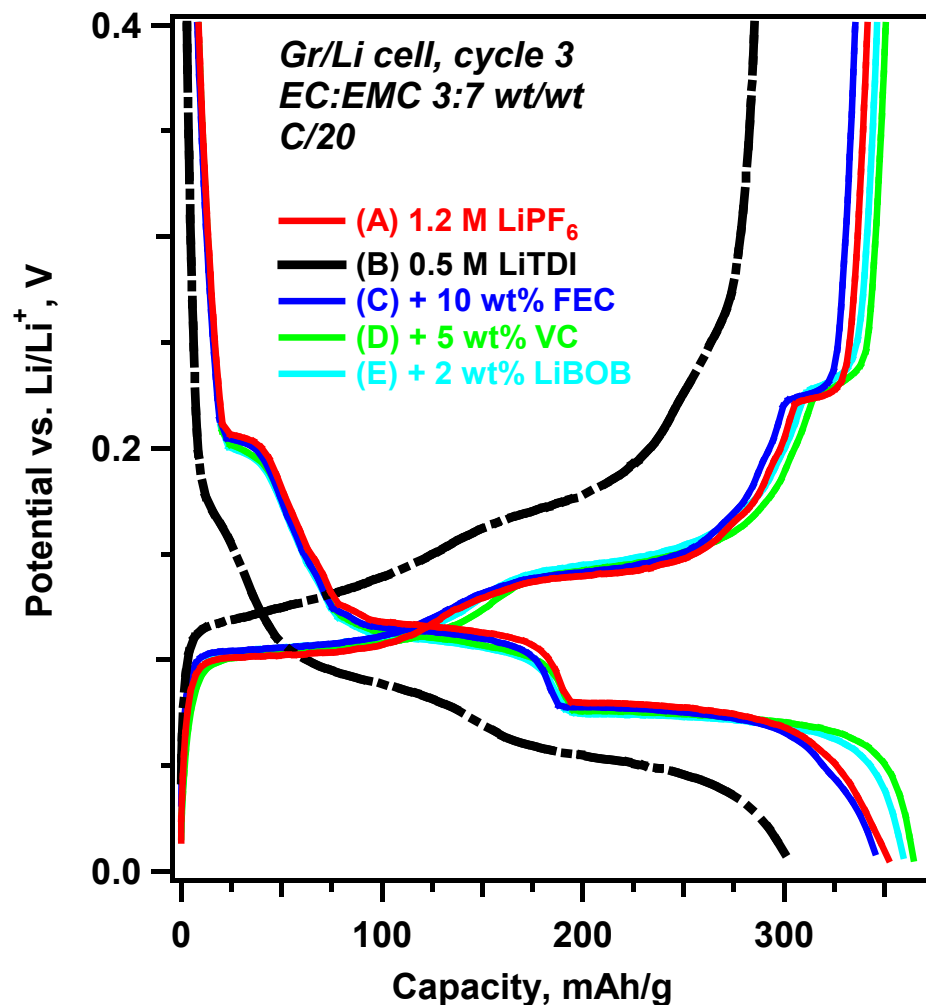
- Electrolyte salts, that could be an alternative to LiPF₆, are being explored
 - Salts, such as LiTFSI and LiFSI, corrode the Al current collector at voltages > 4.0 V vs. Li/Li⁺
 - Our experiments indicate that LiTDI could be used in cells at voltages up to 4.8 V vs. Li/Li⁺



Current vs. potential plots for cells containing 0.5 M LiTDI, 1.2 M LiTFSI, or 1.2 M LiPF₆ salts

GRAPHITE ELECTRODES PERFORM POORLY IN 0.5M LiTDI IN EC:EMC ELECTROLYTE

Cell performance is enhanced by use of electrolyte additives

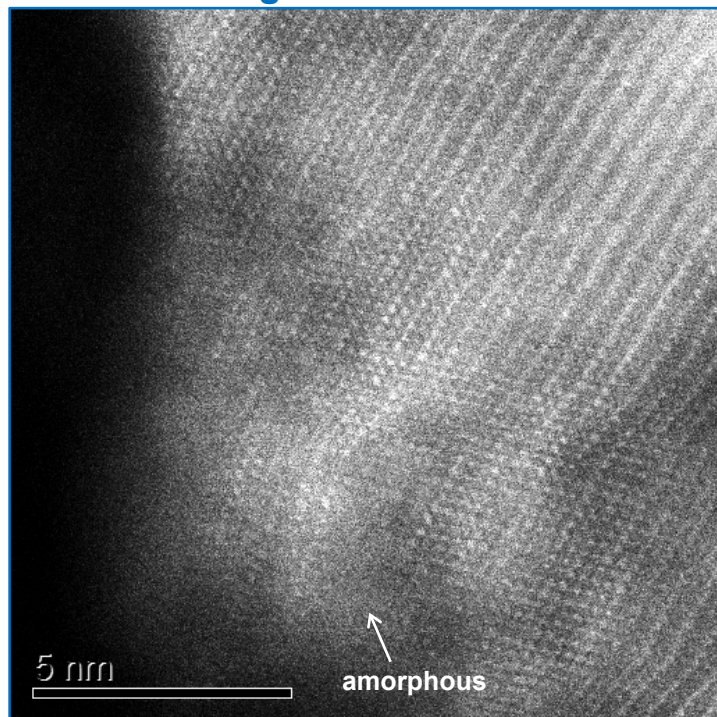


- Graphite/Li cells with LiTDI salt in conventional carbonate solvents show low capacities and high impedance
 - The salt apparently hinders formation of a “good” SEI
 - Addition of compounds, such as FEC, VC, and LiBOB, which are reduced earlier than EC and enhance the SEI, improve cell performance

EXPOSURE OF CATHODE (NMC532) TO MOISTURE CREATES STRUCTURAL CHANGES IN THE OXIDE

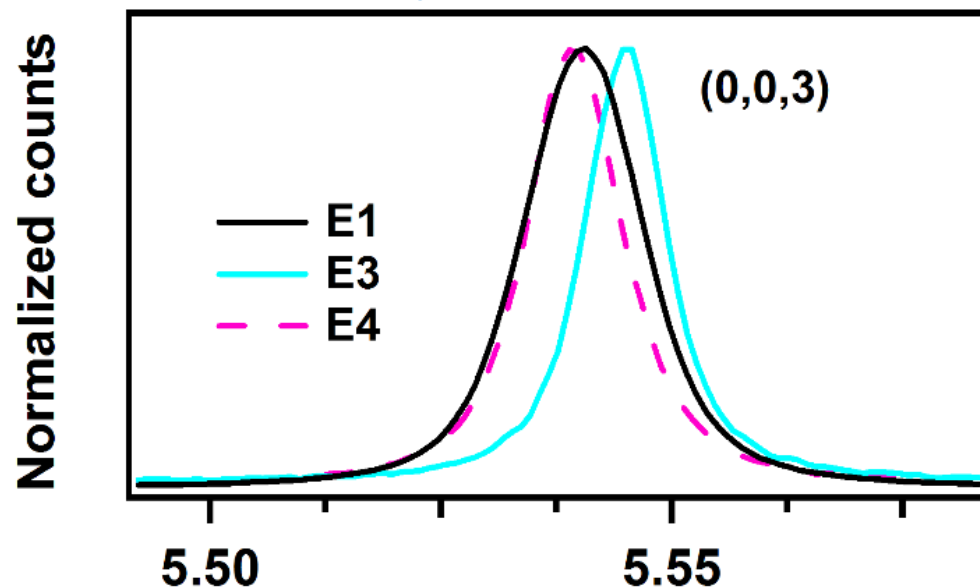
Exchange of Li^+ by H^+ can explain experimental data

STEM image near oxide surface



New phases are seen at basal plane edges. These phases have transition metal ions in Li-planes. Bulk appears to retain layered structure.

Synchrotron XRD data



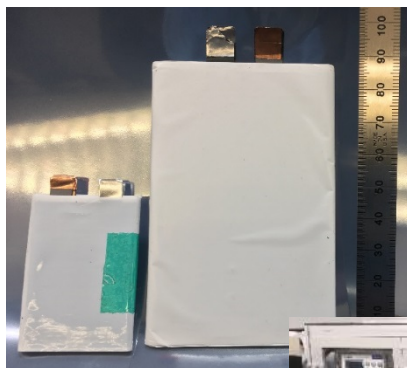
E1: Compared to the pristine sample

E3: (2-month humid-air exposure) shows c-axis contraction (0.06%) and a-axis dilation (0.03%)

E4: (Relithiation of E3) appears to restore crystal structure

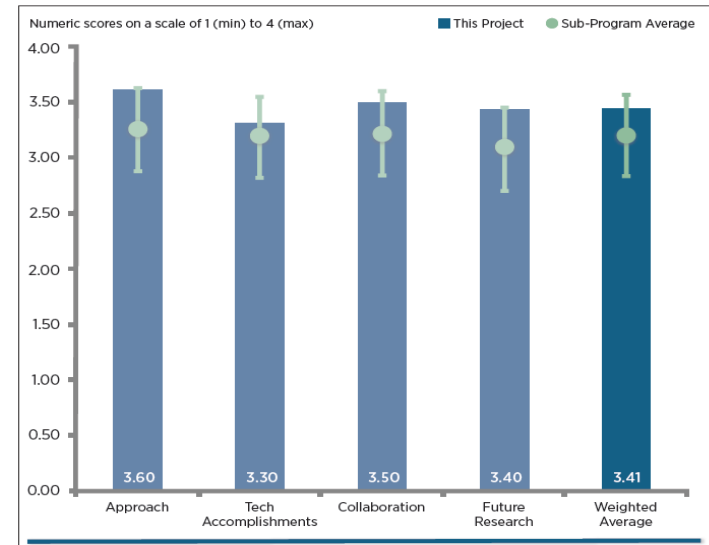
EQUIPMENT UPGRADES FOR CELL FABRICATION

- Installed new Z-fold pouch cell stacker that is capable of making xx3450 (~0.5Ah) and xx6395 (~2 Ah) pouch cells
- Upgraded the 18650 winder for multiple current tabs and electronic alignment
- Installed new Buhler-Primix mixer with dual planetary blades and dispersion blade
- Installed new Anton-Parr rheometer for better mixing control
- Tripling dry room space in FY17-18 (ANL-IGPP & Royalty Funds)



REVIEWER COMMENTS FROM 2016 ANNUAL MERIT REVIEW

- *“The reviewer expressed concern that little was discussed with respect to quality control of the fabrication process. For these scale-up to be impactful, the performance of the tapes and cells have to be truly state of the art....;The reviewer asked how much benchmarking has the project done as compared to commercial products, and would like to know what the details of the quality control are.”*
- We were also concerned about the state-of-the-art status for our equipment and have invested in new pouch cell equipment (Z-fold stacker), upgraded our 18650 winder, purchased a new dual-blade planetary mixer, and a multi-function viscometer. Regarding benchmarking, we always compare our results to a relevant baseline cell system that is usually made and tested in parallel. We document our materials, mixing process, and test conditions so that they may be repeated in the future.



- *“The reviewer noted that there has not been any [Si] breakthrough result as yet, but this is a challenging task around the globe and studies carried out at CAMP will certainly help in better understanding the factors that will help in developing a robust Si electrode.”*
- The CAMP Facility is working closely with the Si Deep Dive effort to speed this process along.

CAMP FACILITY'S ELECTRODE LIBRARY SERVES THE BATTERY COMMUNITY

- The Electrode Library serves as a supply of standard electrode samples that are designed to be interchangeable with one another (capacity matched).
- Electrodes can be made with as little as 50g of experimental material, and can be made to match an existing counter electrode.

	Electrodes Delivered							
	FY14		FY15		FY16		FY17 (Oct-April)	
Argonne	116	13.3%	206	12.2%	174	8.6%	55	4.1%
Other Labs	213	24.4%	373	22.0%	726	35.9%	75	5.6%
Universities	119	13.6%	83	4.9%	117	5.8%	48	3.6%
Industry	423	48.5%	1028	60.8%	1004	49.7%	1157	86.7%
Total	871		1690		2021		1335	

Currently Available:

- 11 anodes
- 22 cathodes
- Neg:Pos balanced
- 2 mAh/cm²
- 220 mm x 110 mm of coating per sheet

CAMP FACILITY: ELECTRODE & CELL FABRICATION COLLABORATORS

Universities



Industry



National Laboratories



REMAINING CHALLENGES/FUTURE WORK

- Fabricate new generation of silicon-graphite electrodes using larger silicon particles
 - 70-130 nm silicon from NanoAmor or Paraclete Energy; move towards 30% Si
 - Optimize silicon-graphite-binder-solvent to make a robust composite electrode
 - Continue to improve silicon-graphite composite electrode quality and gain a better understanding of slurry/electrode properties
 - Fabricate Si-Gr//NMC pouch cells in support of CAEBAT Program at NREL
- Develop techniques for in-operando detection of Li-plating during fast charging
- Continue efforts in determining effect of cycle life/calendar life/cell pressure on silicon-graphite vs. NMC532 pouch cells
- Continue effort on effects of electrode loading (on-going effort started in FY15)
 - Use reference electrodes to determine rate limiting electrode (anode or cathode)
 - Determine influence of segmented charge rates for low, medium, and high SOC
- Continue support of High-Energy/High-Voltage and Next Generation Anodes (Si) Deep Dive Projects with new prototype electrode and cell builds
- Continue work with Post-Test Facility on determining failure mechanisms in Si cells
- Continue work with MERF on demonstrating performance of new materials, including new conductive binders for silicon (with LBNL)
- Any proposed future work is subject to change based on funding levels

SUMMARY

- Fabricated multi-stack pouch cells of 0.5 Ah with 15% silicon and NMC622
 - Preliminary results showed no difference between cycle life and calendar life when comparison based on number of cycles
- Developed silicon-graphite composite electrode trials with new sources of Si
 - Must optimize particle-binder system for better mechanical properties
 - **More Particle Morphology/SEI Formation/Binder Interface work needed!**
- Examined performance of lithium-ion cells with LiTDI electrolytes
 - LiTDI prevents anodic dissolution of Al current collectors
 - LiTDI is reduced on lithiated graphite - yielding a thick and resistive SEI layer
 - Adverse effects of LiTDI reduction can be prevented by electrolyte additives
- Determined voltage-capacity-energy relationship for variety of cathode materials
 - Values needed in BatPac modeling and CAMP electrode loading balancing
- Showed performance of layered oxide (NMC, NCA) electrodes is irreversibly damaged by exposure to moisture due to exchange of Li^+ by H^+
- Upgraded cell fabrication equipment capabilities and began dry room expansion
- Provided High-Energy/High-Voltage Project, Next Generation Anode Project, and other DOE R&D projects with baseline and experimental electrodes and cells

CONTRIBUTORS AND ACKNOWLEDGMENTS

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- | | | |
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| ▪ Shabbir Ahmed | ▪ Alison Dunlop | ▪ Kris Pupek |
| ▪ Khalil Amine | ▪ James Gilbert | ▪ Yan Qin |
| ▪ Dominick Baker | ▪ Manar Ishwait | ▪ Phillip Ridley |
| ▪ Javier Bareno | ▪ Andrew Jansen | ▪ YoungHo Shin |
| ▪ Ira Bloom | ▪ Ozge Kahvecioglu | ▪ Ilya Shkrob |
| ▪ James Ciszewski | ▪ Feridun | ▪ Xin Su |
| ▪ Dennis Dees | ▪ Kaushik Kalaga | ▪ Steve Trask |
| ▪ Trevor Dzwiniel | ▪ Gregory Krumdick | ▪ Pierre Yao |
| ▪ Nancy Dietz-Rago | ▪ Wenquan Lu | ▪ John Zhang |
| ▪ Kevin Gallagher | ▪ Paul Nelson | ▪ Linghong Zhang |

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- Robert Kostecki (LBNL)
- Gao Liu (LBNL)
- Robert Tenent (NREL)
- Chunmei Ban (NREL)
- Claus Daniel (ORNL)
- Jianlin Li (ORNL)
- Gabriel Veith (ORNL)
- David Wood III (ORNL)
- Eric Allcorn (SNL)
- Kyle Fenton (SNL)
- Chris Orendorff (SNL)

Research Facilities

- Materials Engineering Research Facility (MERF)
- Post-Test Facility (PTF)
- Electrochemical Analysis and Diagnostic Laboratory (EADL)
- Center for Nanoscale Materials (CNM)
- Advanced Photon Source (APS)

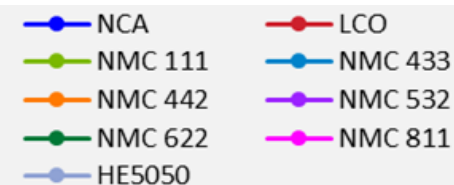
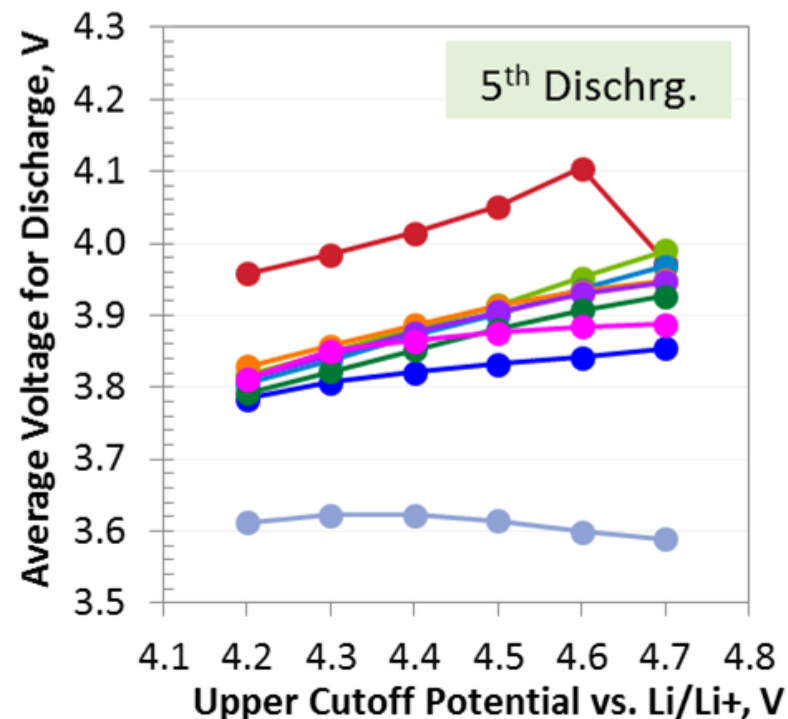
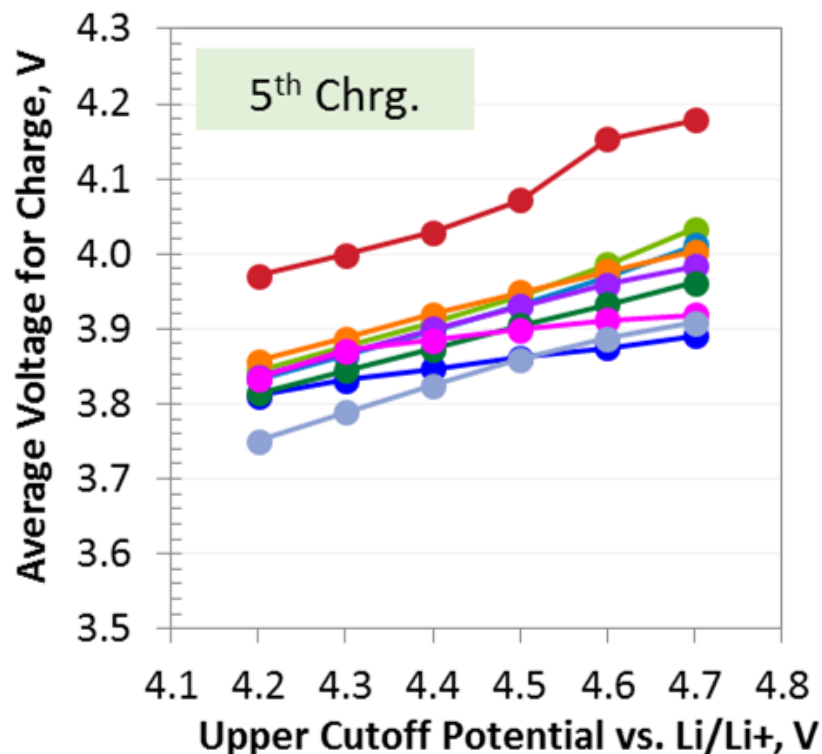
Industry

- Paraclete Energy
- Celgard
- Superior Graphite
- Targray
- Forge Nano

Support from Peter Faguy and David Howell of the U.S. Department of Energy's Office of Vehicle Technologies is gratefully acknowledged.

TECHNICAL BACK-UP SLIDES

VOLTAGE WINDOW STUDY: AVERAGE VOLTAGE, V



- These plots show the trends of average voltages for a ~C/24 cycle
- Average voltages reported in these plots were calculated at end of charge (LEFT) and discharge (RIGHT) for the 5th cycle and averaged
 - $\text{total Wh} \div \text{total Ah} = V$